

## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Appl. No. 10/734,014 Confirmation No.: 2369  
Applicant(s): JOHN G. NUNAN  
Filed: December 11, 2003  
TC/A.U. 1795  
Examiner: Matthew J. Merlding

Title: EXHAUST TREATMENT DEVICE, AND METHODS OF MAKING  
THE SAME

Docket No.: 034166.053  
Customer No.: 25461

Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450  
Sir:

**DECLARATION UNDER 37 CFR 1.132**

John Nunan, inventor named herein, hereby declares and states as follows:

The following describes the experiments carried out under his direction and control to establish characteristics of the subject matter described and claimed in the above identified U.S. patent application.

Reference is made to the eight (8) figures attached hereto.

Figure 1: This shows the performance of a reference 1-layer catalyst as compared to two test catalysts (blue and darker red bars), representative of the invention described in my above-identified patent application. The reference micro-pore catalyst uses conventional materials with micro-pore oxygen storage components (OSCs) and aluminas at a Pt/Rh loading of 25 g/ft<sup>3</sup> and a Pt:Pt:Rh ratio of 3:0:1. These are conventional materials. The results in terms of HC, CO and NO<sub>x</sub> conversion are shown in the first bar on the left in each grouping (red color).

The blue or second bar from the left shows the effect of swapping out the micro-pore OSC for a macro-pore OSC and shows an improvement over the reference 1-layer catalyst. The

App. No. 10/734,014  
Declaration of John G. Nunan

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darker red bar (third from left in each group) shows the added benefit of also including a macro-pore alumina as well as the macro-pore OSC. This combination gives the best performance.

The black bar (last bar in each group on the right) is the reference now loaded with 40 g/ft<sup>3</sup> of Pt + Rh at 5:0:1 ratio and it is seen that even at this higher PGM (precious group metal) loading we do not match the performance of the dark red bar with macro-pore OSC and alumina.

Figures 2 and 3 show further data for the catalysts in Figure 1. We show the COP (cross-over-point) for NO<sub>x</sub>/CO conversion. The higher this value is the better. We also show the integral conversion for CO, HC and NO<sub>x</sub> around stoichiometry for the same catalysts. After the dyno aging and testing was complete the wash coat from these catalysts was removed and analyzed for surface area/porosity. What is presented in the table is the total wash coat surface area, the total pore volume and the pore volume that falls within a pore diameter range of 300 – 1,000 angstroms (meso-macro-pore range). What this data shows is that the COP and NO<sub>x</sub>/CO conversion correlate with pore volume in the range of 300 – 1,000 angstroms BUT does not correlate with total surface area or total pore volume. Thus, what the data also shows is that what we need for maximum performance is not high surface area or total pore volume but pore volume that falls within a certain pore diameter range; i.e. meso-macro pore range. This is a requirement for the high performance of the 1-layer Pd/Rh or other technologies.

Figures 4, 5 and 6. These three figures show different areas of the wash coat that were analyzed using EDX (energy dispersive X-ray). The sample area shown is bombarded with electrons, we remove electrons from lower energy levels in the atoms and electrons from higher energy levels relax or fill the holes with release of characteristic x-rays for the atom in question. The Figures show the areas analyzed; i.e., starting at the top of the wash coat (Figure 4) and

App. No. 10/734,014  
Declaration of John G. Nunan

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progressing through the middle of the wash coat (Figure 5) toward the wash coat at the cordierite surface, Figure 6, and the corresponding EDX spectra. The intensity of the peaks can be used to give a semi-quantitative measure of element concentration. The EDX spectra shows the separate peaks for Pd and Rh; i.e., non-alloyed condition.

In Figures 7 and 8 are shown the semi-quantitative data for the Pd and Rh concentration and concentration ratio through the wash coat. It can be seen that the metals are uniformly dispersed from surface to bottom of the wash coat. Thus, there is no physical separation of the two precious group metals as is a requirement in the *Sung, et al.* patent.

I, John G. Nunan, hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under 18 U.S.C. 1001, and that such willful false statements may jeopardize the validity of the application, any patent issuing thereon, or any patent to which this declaration is directed.

Date:

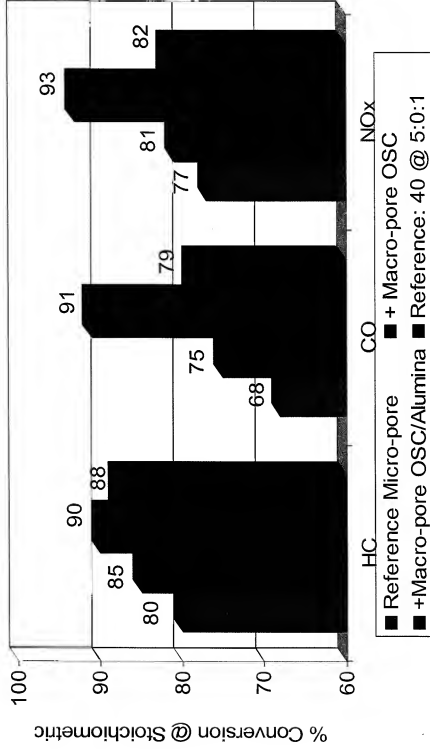
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**Figure 1:** Comparison of 1-Layer/1-Pass technology made with standard micro-pore materials & new 1-Layer technologies made with meso/macro-pore materials. Get much better performance at lower PGM loading compared to reference

**4-Mode-aging w Max. bed T = 1050°C hrs; Pt + Rh = 25g/ft<sup>3</sup> @ 3:0:1 for the reference micro-pore; + macro-pore OSC & +Macro-pore OSC/Alumina; Aging #1**



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**Figure 2:** Comparison of 1-Layer/1-Pass technology made with standard micro-pore materials & new 1-Layer technologies made with meso/macro-pore materials – activity correlates w porosity in the meso-macro range (300 - 1000Å)



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**4-Mode-aging w Max. bed T = 1050°C hrs; Pt + Rh = 25g/ft<sup>3</sup>  
@ 3:0:1 for the reference micro-pore; macro-pore OSC &  
Macro-pore OSC/Alumina; Aging #1**

Sample ID	A/F sweep Test				S A	Pore Volume Cumulative 300 - 1000 Å°	Total Pore Volume Cumulative
	COP	HC	CO	NOx	m <sup>2</sup> /g	cm <sup>3</sup> / g	cm <sup>3</sup> / g
Reference micro-pore.	72.0	80	68	76	45.0	0.063	0.227
Macro-pore/OSC	77.7	84	74	81	57.3	0.165	0.317
Macro pore OSC/Alumina	91.9	87	87	90	45.8	0.195	0.311

**Figure 3:** Comparison of 1-Layer/1-Pass technology made with standard micro-pore materials & new 1-Layer technologies made with meso/macro-pore materials – activity correlates w porosity in the meso-macro range (300 - 1000Å)



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**4-Mode-aging w Max. bed T = 1050°C hrs; Pt + Rh = 25g/ft<sup>3</sup> @ 3:0:1 for the reference micro-pore; macro-pore OSC & Macro-pore OSC/Alumina Aging #2**

Sample ID	A/F Sweep Data				S A	Pore Volume Cumulative 300 - 1000 Å°	Total Pore Volume Cumulative
	COP	HC	CO	NOx			
Reference Micro-pore	77.8	91	72	81	59.1	0.078	0.298
Macro-pore OSC	95.2	95	88	91	57.0	0.135	0.305
Macro-pore OSC/Alumina	99.3	95	90	92	49.7	0.196	0.335

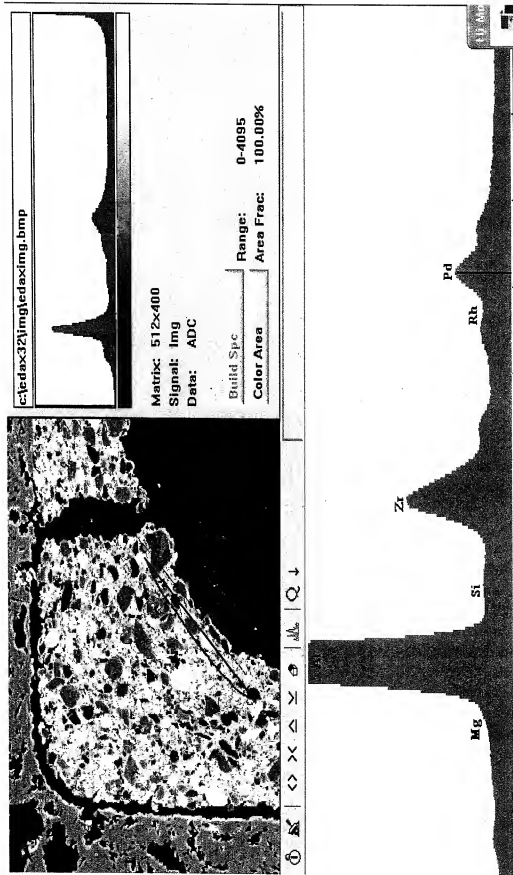
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**Figure 4:** EDX analysis of Pd and Rh concentration as a function of location in WC layer. Analysis done sequentially in locations starting from WC outer surface (Area #1) to cordierite at corner.



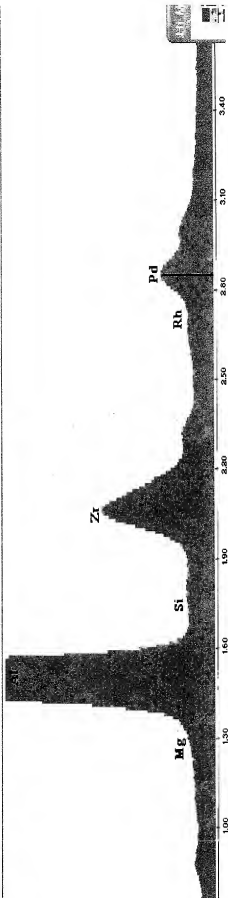
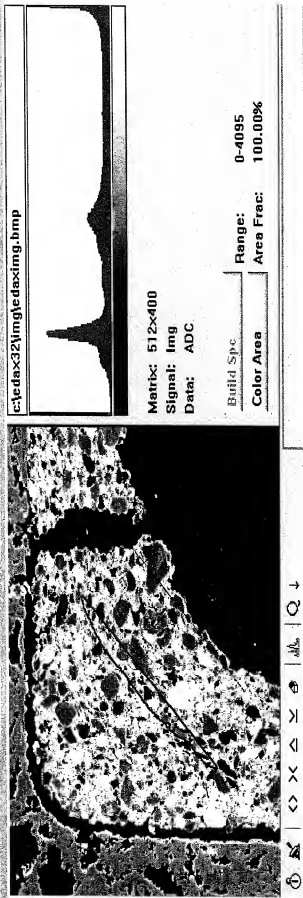
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**Figure 5:** EDX analysis of Pd and Rh concentration as a function of location in WC layer. Analysis done sequentially in locations starting from WC outer surface (Area #7) to cordierite at corner.



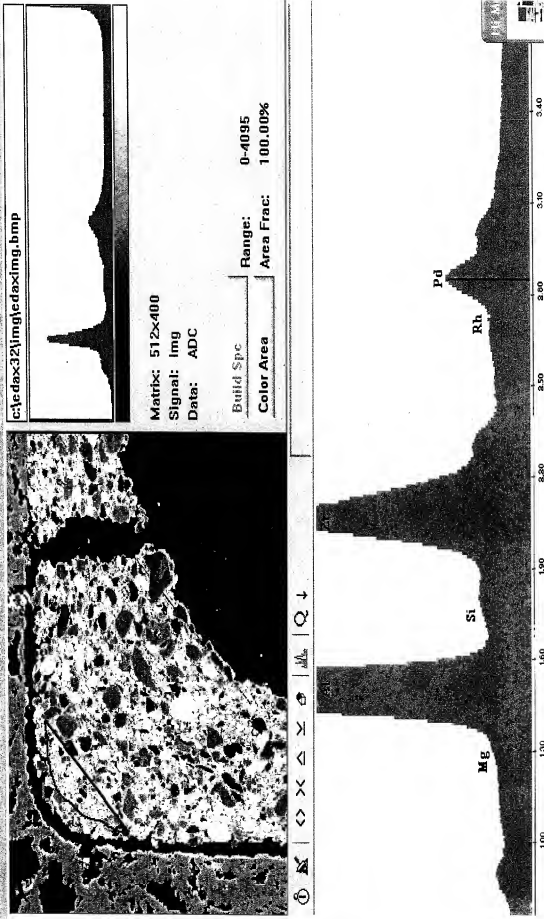
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**Figure 6:** EDX analysis of Pd and Rh concentration as a function of location in WC layer. Analysis done sequentially in locations starting from WC outer surface (Area #12) to cordierite at corner.



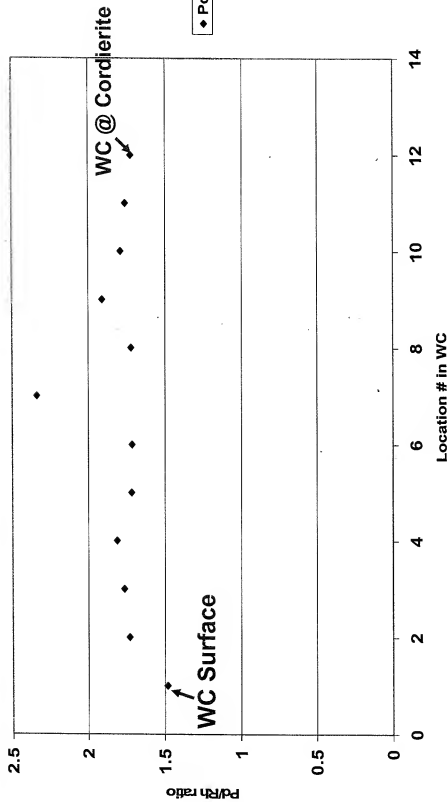
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**Figure 7:** Weight ratio of Pd/Rh as a function of location from WC surface (Location #1) to surface of Cordierite (Location #12) showing that Pd/Rh weight ratio is constant within experimental error

Pd/Rh Ratio as measured by by EDS



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**Figure 8:** Weight% Pd & Rh as a function of location from WC surface (Location #1) to surface of Cordierite (Location #12) showing that Pd & Rh concentration are constant within experimental error

Weight % Pd & Rh as a function of location in WC as measured by EDS

